SOP #BR-0400

Decontamination of Containers and Sample Preparation Equipment

Brooks Rand Labs

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Decontamination of Containers and Sample Preparation Equipment

1.0 DESCRIPTION

1.1 Definition: Removal of any trace element contaminants in sample vessels or laboratory equipment to ultra-trace levels.

1.2 Scope: One of the most important tasks in the laboratory is equipment decontamination. The equipment is used for a variety of tasks, including sample collection, storage of samples for laboratory analysis, sample preparation, and storage of reagents. This protocol will outline the cleaning processes and point out the basic precautions to ensure cleanliness to ultra-trace levels.

1.3 Summary: Once identified as requiring cleaning as outlined in standard operating procedure (SOP) BR-0303, the sample container is emptied of its contents, rinsed and cleaned, and re-rinsed. The container is then either filled with 0.4% hydrochloric acid and stored until use (Teflon® containers), stored in a 10% hydrochloric acid vat (vials, caps, and tubing for distillations and vials, funnels, and marbles for solid preparations), stored in a 30% nitric acid vat (containers used for trace metals analysis by ICP-MS), or stored in their original shipping cases until use (fluorinated polyethylene (FLPE) bottles, glass bottles, and glass vials for mercury work and HDPE bottles for hydride analysis).

2.0 EQUIPMENT AND MATERIALS

2.1 Concentrated hydrochloric acid (HCl), 35-37% (w/v), trace metals or "instra-clean" grade, tested to be < 5 pg Hg/mL and sufficiently low in other trace metals.

2.2 Concentrated nitric acid (HNO_3), 70-71% (w/v), trace metals grade, tested to be < 5 pg Hg/mL and sufficiently low in other trace metals.

2.3 Bromine monochloride (BrCl): In a fume hood, dissolve 27 g of reagent grade KBr in 2.5 L of concentrated instra-clean HCl. Place a magnetic stir bar in the bottle and stir for approximately 1 hour. Then, slowly add 38 g reagent grade $KBrO_3$ to the acid while stirring.

> *WARNING: This process generates copious quantities of free halogens which are released from the bottle. For this reason, it is vital that the $KBrO_3$ is added slowly in a fume hood!*

When all of the $KBrO_3$ has been added, the solution should change from yellow to red to orange. Loosely cap bottle and allow to stir overnight before tightening the lid. All BrCl must be tested prior to use.

2.4 Hydroxylamine solution:DIW with 10% (w/v). hydroxylamine hydrochloride. (100 g of $NH_2OH \cdot HCl$ dissolved into 1 L of DIW).

2.5 Teflon® cylindrical vats (for 50% HNO_3) and rectangular polypropylene vats (for 30% HNO_3 and 50% HCl).

2.6 Heating blankets and immersion heaters.

2.7 Safety gear (See section 3.1 "Safety Gear and Safety" for a full description of necessary safety equipment).

2.8 Concentrated hydrofluoric acid (HF), trace metal grade.

2.9 Deionized water (DIW).

3.0 PROCEDURE

3.1 Safety Gear and Safety: As with all lab work, a lab coat must be worn at all times. The following minimum amount of gear must be worn at all times while working with the either the hydrochloric or nitric acid vats:

- Chemical resistant gloves (as liners)
- Extra-long rubber gloves as an outer layer glove
- Lab coat
- Rubber apron
- Air purifying respirator mask with compatible cartridges
- Eye glasses or goggles
- Face shield

Protective eyeglasses or goggles are not a substitute for the face shield. Both must be worn. This safety gear must be worn to ensure safety and compliance with BRL safety regulations (see the BRL Chemical Hygiene Plan for further details). Protective boots are not required but, due to the possibility of acid drip, are strongly advised.

Safety Notes: *In case of skin contact with acid, wash the exposed area with copious amounts of cold water for a minimum of ten minutes. Sodium bicarbonate ($NaHCO_3$) should be kept near the acid room for neutralizing any acid that may contact the skin. Soda ash should also be kept nearby to neutralize any acid spills. A safety shower is also located next to the acid vat room. All personnel must be aware of the safety shower location and operation. Personnel* ***must*** *receive training prior to working with HF.*

3.2 Acid Vats: Of all the steps involved in the decontamination of vessels, the most potentially hazardous are those where the vessels are placed in and removed from the acid vats. Although serious injury could possibly occur, the probability of injury can be greatly reduced by following common sense precautions as discussed below.

3.2.1 There are three Teflon® vats and six polypropylene vats that are currently used for equipment decontamination. All heated acid vats are kept in the fume hoods inside the decontamination room. The three small Teflon® vats (Vats A, B, and C) contain 50% HNO_3 . The large rectangular vats contain 50% HCl. When in use, the acid vats are heated to 60 ºC ± 5 ºC. Another rectangular vat is used for special projects when required, and is located in the bottle washing room.

The following vats are never heated. The large 90-gallon polypropylene vat in the acid vat room contains 30% HNO_3 and is used for trace metal work only. A second smaller polypropylene vat filled with 30% HNO_3, also located in the acid vat room, is used to clean containers and to clean oven bombs used to prepare sediment samples and other containers that have elevated levels of metals contamination. These container types must never be placed in the larger 30% HNO_3 vat. The third 30% HNO_3 vat is used to store cleaned marbles and is located in the prep lab. Finally, there are three polypropylene vats that are filled with 10% HCl, stored in the prep lab, and are used to store various containers, tubing, and glassware after it has been cleaned.

3.2.2 Trace metal grade acid is used when acid must be added to the vats. Each of the nitric cylindrical vats holds up to 10 liters. Each vat is filled to about ¾ full with a 50% HNO_3 (always add water first before adding the acid). If the vats are filled any deeper, there is a risk of acid running over into the user's gloves or onto the counter when bottles are placed into the vats. When acid is added to a vat, the amount added to each vat is recorded on the log sheet kept in the acid vat room. Currently only the 2.5 mL vials used for MMHg analysis are cleaned in the 50% HNO_3 vat

NOTE: The acid level in the 50% HCl vats must be kept high enough to ensure that the emersion heaters used to heat the vats remain submerged. Failure to do so will cause the vat to melt down.

3.3 Loading the Acid Vats: Only items which are made of HDPE, Teflon®, or glass can be cleaned in the vats. Teflon® bottles can be identified by the blue hue or by the identification stamp "FEP" or “PFA” on the bottom of the bottle.

3.4 Turning on the Heated Acid Vats: Sample containers are loaded into the vats and heated for 12 hours. An electronic timer shuts off the heater at the end of the heating cycle. Vats are then allowed to cool before they can be emptied and/or be reloaded . The 50% HNO_3 vats and the 50% HCl vats are heated at 60 ºC for 12 hours.

3.5 Cooling and Emptying the Acid Vats: The vats are the most dangerous when the acid is hot. The chance of a severe burn is greatly increased, and the gloves cannot withstand prolonged exposure to hot acid. Therefore, NEVER STICK YOUR HAND INTO A HOT ACID VAT. This should never be done even if wearing protective gloves. *Always allow the vats to cool to room temperature*. The 50% HNO_3 vats and the smaller

50% HCl vat usually take approximately 2-4 hours to cool. Due to the large size of the 50% HCl vat, it should be allowed to cool for at least 6 hours. The cooling process can be sped up by removing the lid and/or by removing the heating element from the vat. Removing the lid, however, will release a tremendous amount of acid fumes. Although these fumes are efficiently vented to the outside, removal of the acid vat lids should only be done when sample container needs are urgent.

3.5.1 Be cautious when removing the lids on the vats. After the vats have been heated, acid condenses on the inside of the lid. Always remove the lid slowly to vent the fumes, and allow the condensed acid to drip back into the vat.

3.5.2 After the vats have been heated and cooled for the appropriate length of time, dump any acid in the bottles or container back into the heated vat and transfer the bottles into the rinsing vat, located by the vat. The water in the rinsing vat should be changed each time the vats are emptied to avoid contamination and to ensure that the water does not get too acidic.

3.5.3 Containers may be removed from the acid vat using appropriately gloved hands. Be careful not to splash the acid while removing the vessel because it will ruin equipment and damage clothing. Pour the acid from inside the container back into the vat, and place the empty bottle into the rinsing vat. When removing items from the vat, be careful to prevent acid from spilling over into gloves. The beakers full of bottle lids can then be removed and the acid carefully drained back into the acid vat. Repeat this procedure for each vat and for both types of acids, nitric or hydrochloric. Once the vats are empty, they may be filled again with washed or new vessels. If the vats are not to be used they should be left in the fume hoods in the acid room with lids on and the heat off.

3.5.4 To clean up the vats and acid room, use a damp rag to wipe up any droplets of acid on the surrounding area and then use a dry rag to wipe up the excess water.

3.6 Rinsing and Putting Bottles into the Oven: Always wear vinyl clean-room lab gloves when rinsing the bottles for two reasons: 1) the rinsing vat, which contains water, will become acidic after repeated use and will cause irritation to the skin, and 2) clean-room gloves will prevent contamination of the bottles. Nitrile gloves may be worn beneath the vinyl clean-room gloves for additional protection.

3.6.1 Remove the bottles from the rectangular rinsing vat and wash them under the DIW faucet. Rinse them three times inside and out, filling the vessel and allowing it to overflow the rim each time. The vessel lids should also be rinsed three times.

3.6.2 The outside of the capped vessels should then be rinsed of excess acid, dried in the clean air hood and placed into the oven overnight at a temperature of

about 55 °C. The next day the oven should be turned off and the bottles removed. The acid solution used for this step is then emptied, neutralized and disposed.

3.6.3 Bottles should then be triple rinsed again. Teflon® bottles are acidified with 0.5 mL HCl per 125 mL (0.4%), bagged, and stored. At this stage, all bottles receive the same instra-clean HCl for acidification.

3.7 Processing of Dirty Bottles: Two months following the reporting of final results to the client (or as dictated by the contract), the contents of the original sample container can be disposed. Several of the reagents used in the mercury analysis are hazardous, and produce fumes that may be harmful, causing such immediate health problems as headaches and sore throats. One of these reagents, bromine monochloride, can be identified by its yellow color and distinctive odor and it must be reduced with 10% hydroxylamine solution before further disposal. Sample disposal procedures are located in BR-0303 and must be followed. Goggles and gloves should be worn at all times. Upon sample disposal, the sample container may begin going through the decontamination process if the container is to be reused.

3.7.1 All sample vessels to be reused must be washed with a brush, Alconox® or 409®, and deionized water prior to being put through the acid vats (HNO_3 or HCl). Water in the sample vessels will bead-up if there is no residue. When rinsing sample bottles, if the water does not bead-up, the bottle(s) must be rewashed until the water beads-up during rinsing.

3.7.2 Containers are separated according to the resulting total mercury or trace metals concentrations (R) of the sample. Using the R-values for total mercury, vessels are categorized as follows; they are:

- $0.1 > R$
- 0.5 ng/L $\geq R \geq 0.1$ ng/L
- 50 ng/L $> R > 0.5$ ng/L
- 100 ng/L $> R \geq 50$ ng/L
- $R \geq 100$ ng/L

Trace metals containers may be reused as long as they are below the element specific criteria shown in Table 2. The cleaning process of the containers is determined by which category the vessels fall within. The cleaning process is explained in section 3.8.

3.8 Bottle Specific Requirements for Reusable Containers: The following cleaning procedures are for all containers that are not routinely disposed of after one use or require special cleaning procedures prior to use.

3.8.1 *Used Teflon® Bottles*: The following guidelines are used to determine the level of cleaning necessary for bottles that contained water samples.

3.8.1.1 Bottles that contained water samples with total mercury concentrations **(R) ≤ 0.1 ng/L** can be cleaned by rinsing three times with DIW then re-acidified with 0.4% instra-clean HCl.

3.8.1.2 If **0.5 ng/L > R > 0.1 ng/L**, the bottle must be rinsed three times with DIW, acidified with 0.8% HCl (0.8% $NH_2OH{\cdot}HCl$ may also be used), placed in the oven overnight, rinsed three times with DIW, and re-acidified with 0.4% of instra-clean HCl.

3.8.1.3 If **50 ng/L > R ≥ 0.5 ng/L**, the bottle must be washed with Alconox® or 409®, rinsed with DIW, immersed in the 50% HCl vat for 24 hours, rinsed with DIW, acidified with 10% HCl ($NH_2OH{\cdot}HCl$ may also be used), placed in the oven overnight, rinsed with DIW, and then re-acidified with 0.4% instra-clean HCl.

3.8.1.4 If **100 ng/L > R ≥ 50 ng/L**, the bottle must be washed with Alconox® or 409®, rinsed with DIW, immersed in the 50% HNO_3 vat for 24 hours, rinsed with DIW, immersed in the 50% HCl vat for 24 hours, rinsed with DIW, acidified with 10% $NH_2OH{\cdot}HCl$, placed in the oven overnight, rinsed with DIW, and then re-acidified with 0.4% instra-clean HCl.

3.8.1.5 If the **R ≥ 100 ng/L** the bottle must be washed with Alconox® or 409®, rinsed with DIW, immersed in the 50% HNO_3 vat for 24 hours, rinsed with DIW, immersed in the 50% HCl vat for 24 hours, rinsed with DIW, acidified with 10% $NH_2OH{\cdot}HCl$, placed in the oven overnight, rinsed with DIW and re-acidified with 10% $NH_2OH{\cdot}HCl$, again placed in the oven overnight, rinsed with DIW, and then re-acidified with 0.4% instra-clean HCl. Figure 1 illustrates the cleaning process of Teflon® bottles.

NOTE: If the bottles are extremely dirty (i.e. stained or used for high level samples) they should be cleaned in the 50% HNO_3 vat for 12 hours followed by immersion in the 50% HCl vat for 12 hours. During the cleaning process, it is best to separate the bottles that contained hazardous levels of mercury away from other bottles to prevent contamination.

3.8.2 *New Teflon® Bottles*: All <u>new</u> Teflon® bottles must be placed in the 50% hydrochloric acid vat and heated for no less than 24 hours. After heating, the vat is turned off, cooled, and later emptied. New tubing, vials and other Teflon® items used for distillations are cleaned for 24 hours in the 50% HNO_3 vat.

3.8.3 *Extraction Teflon® Bottles and Vials*: All Teflon® bottles and vials used in the analysis of methyl mercury (MMHg) by extraction in DCM (water and sediments) must be:

- Scrubbed with Alconox until no stains are visible.

  NOTE: The use of a small amount of DCM or other organic solvent may be necessary to remove stubborn stains from some bottles. This solvent must be recycled until no longer effective. Used solvent must be disposed of in accordance with state and federal laws regulating the specific solvent used. All use of DCM must take place within the confines of a properly working fume hood.

- Rinsed thoroughly with DIW
- Filled with 1.6% BrCl in DIW (2 mL BrCl into 125 mL DIW) and allowed to sit overnight. This step must be performed in the fume hood.
- Triple rinsed with DIW,
- Filled with a solution of 10% NH_2OH-HCl (@1mL/250 mL) and placed in the oven overnight
- Triple rinsed with DIW.
- Filled with a solution of 2% HCl and placed in the oven overnight.
- Tripple rinsed with DIW
- Filled with 0.4% HCl, capped and double-bagged for storage. The cleaned vials are stored in the 10% HCl vat.

Figure 1 summarizes the cleaning process for extraction bottles.

3.8.4 *Water Teflon® Vials, Tubing, and Caps*: Vials, tubing, and caps used for water samples are as follows:

3.8.4.1 For vials, tubing, and caps used to distill any sample with a final methyl mercury result of **10 ng/L** or less:

- Washed with Alconox® or 409® and rinsed with DIW.
- Filled with a 2% HCl solution. The tubing and caps are also injected with the 2% solution. (Note: This step must be performed in a fume hood.)
- The vials are capped with the tubing firmly in place and are heated in the oven overnight at temperatures of 55-60 °C.
- They are then triple rinsed with DIW and placed in the room temperature 10% HCl vat for storage until use.

3.8.4.2 For vials and tubing used to distill high-level samples (**R > 10 ng/L**), additional cleaning is required.

- Washed with Alconox® or 409® and rinsed with DIW.
- Filled with a 2% BrCl solution (caps, tubing, and vials), capped with the tubing firmly in place, and stored on the counter overnight. Then they are triple rinsed with DIW.
- Filled with a 2% solution of NH_2OH-HCl in DIW (caps, tubing, and vials), capped with the tubing firmly in place, and heated in the oven overnight at 55-60 °C. Then they are triple rinsed with DIW.
- Filled with 2% HCl solution (caps, tubing, and vials), capped with the tubing firmly in place, and heated in the oven overnight at 55-60 °C. Then they are triple rinsed with DIW.
- Finally, they are placed in the room temperature 10% HCl vat for storage until use.

3.8.5 *HDPE (High Density Polyethylene) bottles for Trace Metal Analysis*: New HDPE bottles are cleaned to be used for trace metals analysis as follows.

- Soak in cold 30% HNO_3 for 48 hours (Make sure bottles are completely submerged)
- Rinse 3 times with generous amounts of DIW
- Leave in a class 100 clean air station uncapped until dry
- Submit 10% of the cleaning lot for testing.
- Place the rest of the bottles in a plastic bag and deposit in the store room until test results are available.

3.8.6 *Cleaning Process for Glass "APDC" (250ml digestion bottles) and caps, 60ml Teflon® digestion "bombs" and 15ml Teflon® vials (used for APDC and RP preparations)*: APDC jars, caps, 60 ml Teflon® bombs and vials are cleaned as follows:

- Rinse with generous amounts of DIW
- Thoroughly scrub the 60 ml bombs with 409® or similar cleanser known to be low in trace metals before sinking in any of the vats.
- Soak in the cold 30% HNO_3 vat for 48 hours (*Note: APDC jars and vials (15 mL) should be placed in the low-level 30% HNO_3 vat in the bottle washing room and the 60 mL bombs must be placed in the high-level 30% HNO_3 vat located in the sample preparation laboratory.*)
- Rinse and fill with a 0.5% HNO_3 solution.

3.9. Storing the Clean Sample/Preparation Bottles and Vials: After the bottles and vials have gone through the cleaning process and have dried in the clean air hood, containers are double bagged by size and assigned a cleaning batch number, which is the Julian date preceded by the last two digits of the year (i.e. 06-125). The cleaning batch number is marked on the outer bag with permanent ink. After 10% of the bottles or vials have been blank tested (described in Section 4 of this document) and have been determined to pass the acceptance levels (see Table 1 and Table 2 for acceptance levels), bottles are

individually double bagged with the bottle ID (if applicable) and the cleaning batch number (Julian date) on the bag. Individual double bagging of 125 mL bottles and vials is unnecessary. As many 125 mL bottles that fit together are double bagged with the batch number (Julian date) on the outer bag. Similarly, Teflon® vials or bombs of the same size (2.5 mL, 18.2 mL, 25.6 mL, and 60 mL) can be double bagged together.

3.10 Cleaning Sample Collection Equipment for Mercury Deposition Network (MDN) Projects: The following cleaning procedures are to be followed for both new equipment and for equipment returned from the field.

3.10.1 *Cleaning the Thistle Tubes and Funnels*: The glass thistles and funnels used to collect samples collected as part of the MDN are cleaned as follows.

- Rinse the thistles and funnels and place in the 30% nitric acid vats for 48 hours.
- Remove and rinse thoroughly with DIW and place under clean hood to dry.
- Double bag the thistle tubes individually in zip lock type bags. Then wrap the thistle tubes in bubble wrap to prevent breakage.
- Double bag the funnels individually in zip lock type bags. Then wrap the funnels in bubble wrap.

3.11 Cleaning Carboys

3.11.1 *Standard Procedure for Cleaning Carboys or Reagent Water Containers*: When carboys (or containers) come back in from the field or when new carboys are received from the manufacturer, they must be cleaned as follows:

- Rinse the carboy three times with reagent water and fill with 0.8% (v/v) HCl.
- Allow the carboy to sit on the counter for 3 days, occasionally inverting the carboy so that all inside surfaces come into contact with acid solution.
- After 3 days, empty the carboy and rinse three times with reagent water.
- Every carboy must be blank tested prior to shipping to the client. The carboy must be filled with DIW and allowed to sit overnight. Then 250 mL of the DIW must be collected in a clean FLPE bottle (for Hg testing), a cleaned HDPE bottle (trace metals testing), or both, depending on the analytes of interest to the client.
- Dry the carboy in a clean hood and then double bag. Label the bag with the cleaning date and store in the stockroom.
- The carboy is now ready to be filled and sent out when needed. Carboys should only be used if they have been cleaned in the past 6 months.

NOTE: The Project Manager is responsible for notifying the Sample Control Group and the Trace Metals Group Lead as soon as possible when a carboy will be needed for trace metals analysis to

ensure that there is time to test it for all analytes of interest before shipping it to the client.

3.11.2 *Alternative Quick Cleaning Procedure for Carboys or Reagent Water Containers*: This alternative procedure may be used when carboys need to be turned around quickly for shipping out. As always, a carboy blank must be collected and tested prior to shipping the carboy out.

- Rinse the carboy three times with reagent water and fill 10% full with 10% (v/v) HCl.
- Shake the carboy vigorously for a minute or so. Leave the carboy on the counter, and come back to shake it every 15 minutes or so throughout the day.
- After several hours of shaking, empty the carboy and rinse three times with reagent water.
- Fill the carboy with reagent water, double bag, and label the bag with the cleaning and fill date. The carboy is now ready to send out.

- Testing New FLPE Bottles, Glass Bottles, and Glass Vials for Mercury Work: When new lots of FLPE bottles, glass bottle, and glass vials are received, they must be tested prior to being put in to service, as described in SOP BR-0306.

3.12 Cleaning Glass Marbles:

The marbles are first washed in alconox or 409 solution then rinsed and placed in a 10% BrCl solution for 48 hours, then rinsed and stored in the 30% HNO_3 Vat III, located in the Prep Lab.

4.0 SPECIALTY CLEANING METHODS

4.1 For Cleaning HDPE Bottles that will be Used with an HF/HNO_3 Prep:

- New HDPE bottles are first cleaned as normal (rinsed with reagent water then soaked in 30% (v/v) HNO_3 vat at room temperature for 48 hour).
- The bottles are then rinsed 3 times and filled with reagent water. The bottles for the HF prep should be segregated and identified as their own cleaning lot number at this point.
- Each bottle is acidified to 0.2% (v/v) HF + 0.8% (v/v) HNO_3 [in other words, preserve to 1% with the 20:80 mix of HF:HNO_3]. This acid mixture is stored in the Prep Lab where the TM Group acids are stored.
- Cap each bottle tightly and place the bottles the Class-100 clean-air station until dry.
- Then place the bottles in an oven at 70 °C for 8-24 hours.
- Following oven heating, re-rinse each bottle 3 times with reagent water and place in the Class-100 clean-air station until dry.

10% of the bottles should be filled with 1% (v/v) HNO_3 and taken to the TM Lab for testing. The remaining bottles should be tightly capped and stored in double-Ziploc bags for future use. Label the bottles "For Use with the HF/HNO_3 Prep Only" and list the cleaning lot #.

4.2 Cleaning and Testing Syringes:

- Soak the syringes in 30% (v/v) HCl at room temperature for 48 hours.
- The syringes are then thoroughly rinsed with reagent water and put under a clean air hood to dry.
- Syringes are individually double-bagged.

Testing is done on selected number of the total cleaning lot. To collect samples for testing, the syringe is rinsed with DIW from the DIW faucet and the rinsate is collected in an FLPE bottles. The sample is preserved with 1% BrCl and submitted for Hg testing.

5.0 QUALITY ASSURANCE

5.1 Labeling: All containers holding bottles, vials, or other items being cleaned must be clearly labeled as to what step in the cleaning process the items are currently in. This is to prevent the inadvertent use of items which are not fully cleaned.

5.2 Bottle blanks: Bottle blanks are always performed on every cleaning batch of bottles and vials and the results are recorded in an Excel spreadsheet. Before analyzing bottle blanks, each cleaning batch is quarantined in the storage bins and no bottles are put into regular service. Glassware, Teflon® tubing, MMHg distillation vials, MMHg sediment extraction vials, Teflon® vials for trace metal work, APDC jars, and Teflon® bombs are generally put into service without testing. Teflon® jars and vials, as well as single use glass sample bottles, must be bottle blanked as well. For bottles and vials a minimum of 10% of each type and size class from every cleaning batch is analyzed for total mercury and/or trace metal contamination.

IMPORTANT: All bottles and vials to be blank tested for mercury analyses must be filled with deionized water (DIW) and then acidified (1% pretested BrCl). Bottles used for trace metals work are filled with DIW and acidified to 1% HNO_3.

If a bottle or vial fails to meet the acceptance criterion (See Tables 1 and 2) within a size class, at least two more bottles/vials within that size class are removed from quarantine and tested. If one of these bottles or vials fails as well, then the entire size class of bottles/vials from that cleaning batch is rebatched for decontamination. If the additional bottles/vials pass the acceptance criterion, then only the failed bottle/vial is rebatched for additional cleaning. If all the bottles or vials within a size class pass the acceptance criterion, then the entire size class is released for use.

5.3 Acid Vat Testing: As an added safeguard to ensure that bottles will be decontaminated sufficiently, all acid vats used for cleaning and storage should be tested monthly. The 30% nitric acid vats I & II should be tested for Hg and ICP-MS (Vat II is tested for ICP-MS only). All the other vats are tested for Hg. If any acid vat concentration exceeds the limits set forth in Table 2, then the acid from that vat should be transferred into waste drums for disposal. Specific element limits for the Trace Metals nitric vat are currently being developed. Preliminary limits can be found in Table 2.

6.0 TABLES AND FIGURES

Table 1. Mercury acceptance levels for bottle and vial blanking.

Bottle or Vial Size	Hg Acceptance Level[1, 2]
Teflon, FLPE, or Glass Bottles	< 0.2 ng/L
20-mL & 40-mL Glass Vials	< 0.5 ng/L
2.5-mL, 18.2-mL, & 25.6-mL Teflon Vials (for MMHg only)[3]	< 10.0 ng/L

[1] All equipment must meet the acceptance criteria set forth by the client in the contract prior to any use for that specific project. Contract specific requirements always have precedence over the acceptance criteria set by BRL.

[2] The specific acceptance criteria for any bottle or vial used in work that is covered by DoD QSM, Version 3 requirements is that the analyte of interest is < ½ the analyte specific MRL.

[3] Teflon vials to be used for mercury sample preparation/analysis must be tested and yield results < 0.5 ng/L.

Table 2. Trace metal control limits for bottle blanks and contamination limits for bottles.

Element	Max. Bottle Blank Concentration For 1% HNO_3 [1,2]	Vat Acid Limits	Units
Ag	0.03	1000	µg/L
Al	1.5	15,000	µg/L
As	0.2	3000	µg/L
B	1	600,000	µg/L
Ba	0.05	6000	µg/L
Be	0.05	3000	µg/L
Ca	30	3,000,000	µg/L
Cd	0.01	1000	µg/L
Cr	0.15	3000	µg/L
Cu	0.2	3000	µg/L
Fe	5	30,000	µg/L
Hg	0.2	50,000	ng/L
K	4	600,000	µg/L
Mg	3	150,000	µg/L
Mn	0.05	6000	µg/L
Mo	0.02	6000	µg/L
Na	15	3,000,000	µg/L
Ni	0.2	3000	µg/L
Pb	0.05	1000	µg/L
Sb	0.02	1500	µg/L
Se	0.3	30,000	µg/L
Tl	0.01	600	µg/L
Zn	0.2	8000	µg/L

[1] The specific acceptance criteria for any bottle or vial used in work that is covered by DoD QSM, Version 3 requirements is that the analyte of interest is < ½ the analyte specific MRL.

[2] Teflon vials to be used for mercury sample preparation/analysis must be tested and yield results < 0.5 ng/L.

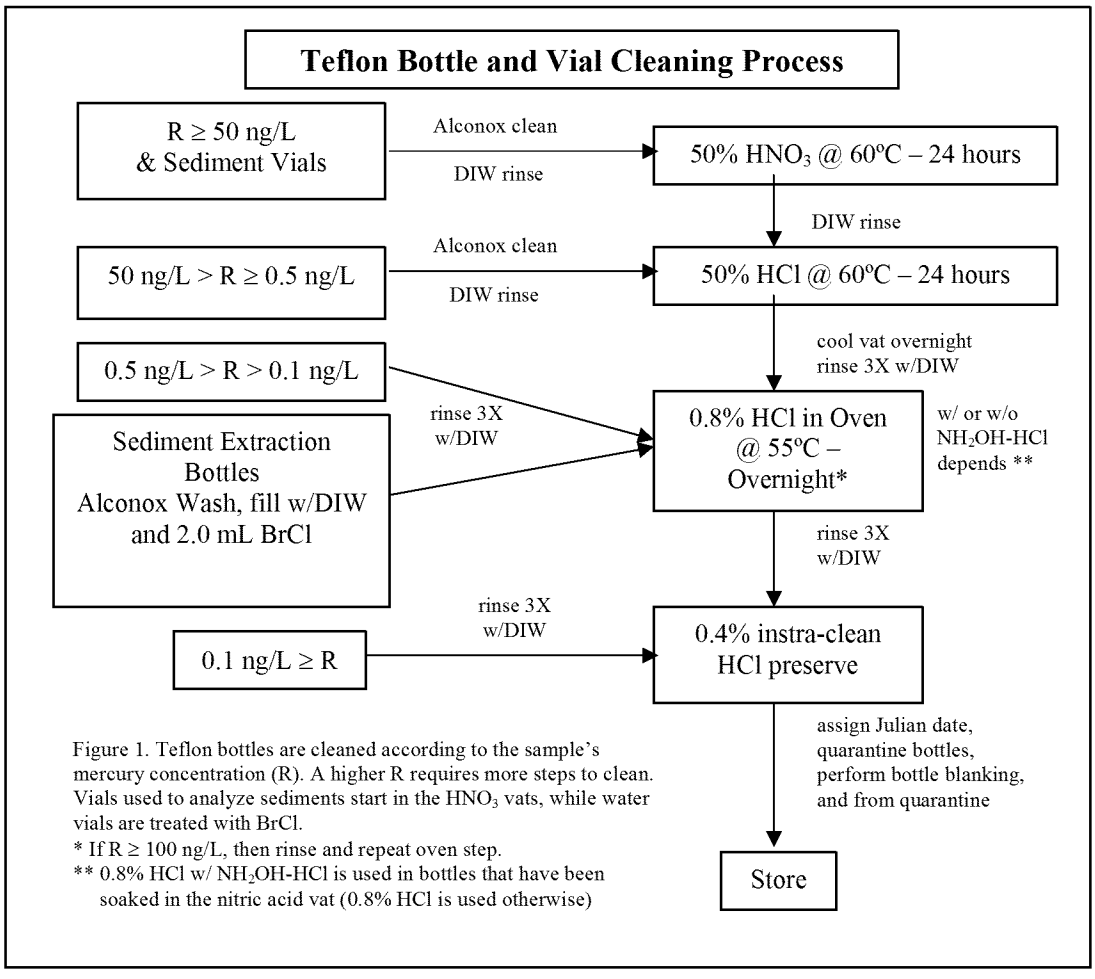

Figure 1. Teflon bottles are cleaned according to the sample's mercury concentration (R). A higher R requires more steps to clean. Vials used to analyze sediments start in the HNO_3 vats, while water vials are treated with BrCl.
* If R ≥ 100 ng/L, then rinse and repeat oven step.
** 0.8% HCl w/ NH_2OH-HCl is used in bottles that have been soaked in the nitric acid vat (0.8% HCl is used otherwise)